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ASD TECHNICAL REPORT 61-203

THE MECHANICAL PROPERTIES OF TANTALUM WITH SPECIAL REFERENCE TO THE DUCTILE-BRITTLE TRANSITION

M. A. ADAMS
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MATERIALS RESEARCH CORPORATION

AUGUST 1961

AERONAUTICAL SYSTEMS DIVISION

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DIRECTORATE OF MATERIALS AND PROCESSES
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FOREWORD

This report was prepared by Materials Research Corporation under USAF Contract No. AF 33(616)-7173. This contract was initiated under Project No. 7351 "Metallic Materials", Task No. 73521 "Behavior of Metals". The work was administered under the direction of Lt. B.A. Wilcox, Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division.

This report covers the work conducted from April 1, 1960 to April 1, 1961.

ABSTRACT

Tensile tests have been made on specimens of commercial tantalum of different grain-sizes, and the variation of lower yield stress σ_y with grain-size 2d has been used to assess the effect of temperature (23°C, -78°C and -196°C) and strain-rate (1.09×10^{-3}/sec. and 9.96×10^{-2}/sec. at each temperature) on the parameters σ_i and k_y in a Petch type equation $\sigma_y = \sigma_i + k_y . d^{-1/2}$. The results indicate that the sensitivity of the yield strength to temperature and strain-rate arises almost entirely from the effects that these variables produce on σ_i which is in agreement with observations for other body-centred-cubic transition metals. The high resistance of tantalum to brittleness has been confirmed, all of the specimens showing pronounced necking and predominantly fibrous fractures; however, on the fracture surfaces of the coarser grained specimens tested at -196°C (both strain-rates) some cleavage facets were observed. This observation of cleavage can be adequately explained by Cottrell's transition equation $(\sigma_i . d^{1/2} + k_y) k_y = \beta \mu \gamma$ when account is made of the effect of deformation on the parameters in the equation, and a value of the effective surface energy for fracture γ is obtained (1.35×10^4 erg.cm^{-2} $\geqslant \gamma > 1.16 \times 10^4$ erg.cm^{-2}) which is in the same range as those values which have been previously measured for niobium, molybdenum and mild steel. The greater ductility of tantalum relative to these materials is attributed to a low value of k_y and a high value of the shear modulus μ. Little or no twinning was observed in the tests, probably because the material was not of a very high purity.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

W. J. Trapp

W. J. TRAPP
Chief, Strength & Dynamics Branch
Metals and Ceramics Laboratory
Directorate of Materials & Processes

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

THE MECHANICAL PROPERTIES OF TANTALUM WITH SPECIAL REFERENCE TO THE DUCTILE-BRITTLE TRANSITION

I. INTRODUCTION:

The introduction of the refractory metals into increasingly greater usage as structural materials, has led in recent years to the stimulation of a new interest in the properties of transition metals with the body-centred-cubic crystal structure. In the area of crystal plasticity a large part of this interest has been centered upon the transition from ductile to brittle behaviour which all these metals exhibit, and this subject has attracted further attention because new quantitative theories of the transition phenomenon have recently been proposed (1)(2). Assessment of these theories, at the time of their publication, was confined primarily to an examination of experimental data for iron and steel, suitable results for other body-centred-cubic transition metals being almost completely lacking. This situation has now, however, been partially rectified by research on niobium (3)(4)(5)(6), chromium (7) and molybdenum (8). The work described in this report expands the scope further, to include experiments on the mechanical properties of tantalum, a material of particular interest because of its reported (9)(10) resistance to brittleness. The observation, by Barrett & Bakish (11), of cleavage facets on tantalum samples hammered to fracture at liquid nitrogen temperature, represents the sole published account of brittle behaviour in tantalum.

To appreciate the significance of the experimental approach adopted in this work it is necessary to critically examine the recent brittle fracture theories (1)(2). These theories stem from two basic ideas; firstly, that cleavage failure is always preceded by plastic deformation; secondly, that crack propagation is a more difficult stage in the cleavage process than crack nucleation. Accepting these ideas one is then led directly to the conclusion that fracture behaviour of a material will depend only on the relative values of the yield stress, and the crack propagation stress for that material under the operating conditions. When the yield stress is smaller than the propagation stress the material

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should be ductile; when it is larger the material should be brittle.

Applying these concepts to a proposed mechanism in which cracks are nucleated from intersecting orthogonal slip bands in the body-centred-cubic lattice, Cottrell(1) derives from energy considerations, an equation

$$(\sigma_i d^{1/2} + k_y) k_y \geqslant \beta . \mu \gamma \tag{1}$$

describing conditions under which a crack nucleus will grow into a full brittle failure. Here, 2d is the grain diameter, μ the shear modulus, γ the effective surface energy for crack propagation and β a constant equal to unity for conventional tensile tests, and 1/3 for tests on notched specimens. σ_i and k_y are parameters in an equation

$$\sigma_y = \sigma_i + k_y . d^{-1/2} \tag{2}$$

originally developed by Petch (12) to describe the dependence of lower yield stress σ_y on grain-size 2d for polycrystalline materials (such as the body-centred-cubic transition metals) which yield discontinuously because their dislocations are locked by impurity atom atmospheres. In such materials the propagation of a Lüders band at the lower yield stress is believed to proceed by a proceess in which the stress fields of dislocations piled-up against grain-boundaries in yielded grains, activate Frank-Read sources in adjacent unyielded grains. For yielded grains of diameter 2d the average pile-up length will be d, and with an applied stress σ_y, and a friction stress σ_i resisting the movement of dislocations across the slip plane, the effective stress acting on the pile up will be ($\sigma_y - \sigma_i$). The formula developed by Eshelby, Frank and Nabarro (13) gives the stress at some distance ℓ ($\ell \ll d$) in an unyielded grain ahead of the pile-up as $(\sigma_y - \sigma_i)(d/\ell)^{1/2}$. A Frank-Read source at distance ℓ, which requires a stress σ_D to release it from its atmosphere, will operate then, when $(\sigma_y - \sigma_i)(d/\ell)^{1/2} = \sigma_D$. This, rearranged and with $k_y = \sigma_D \ \ell^{1/2}$ becomes Eq.(2).

Cottrell's tranistion formula (Eq. (1)) was derived for the specific case of crack nucleation at intersecting orthogonal slip bands. The calculations on which it is based are, however, quite general and should produce similar formulae for the other processes in which glide dislocations are converted into cavity dislocations. Petch (2), using the same basic concepts as Cottrell but a different detailed approach to the brittle fracture problem, arrives aa the same formula with the constants slightly altered.

The importance of Eq. (2) in relation to the brittle fracture theories is largely a practical one. Using this equation one can, by measuring lower yield stress as a function of grain-size, obtain experimental values of σ_i and k_y. Since for a given material, $\beta\mu\gamma$ may be reasonably assumed to remain insensitive to experimental conditions (temperature and strain-rate) it is, according to Eq. (1), changes in σ_i and k_y (with grain-size fixed) which should produce the transition from ductile to brittle behaviour.

In the present experiments measurements of tensile yield stress on specimens of different grain-sizes have been used to determine the effect of temperature and strain-rate on the values of σ_i and k_y for commercial tantalum. The results have been analysed on the basis of Cottrell's transition equation to determine whether the equation adequately describes the fracture behaviour observed in the experiments. An examination has also been made for twinning in tantalum.

II. EXPERIMENTAL PROCEDURE:

A batch of 15" long, 1/8" dia. rods of 99.9% pure tantalum was obtained from the Kawecki Chemical Corporation. All of the rods were prepared from the same 3" dia. sintered billet by successive cold working (rolling and swaging) and annealing (1100°C) treatments, with a 75% cold swage as the final forming operation. The nominal impurity content of the starting material, as quoted by the supplier, is given in Table I.

Recrystallization of the rods was achieved by electron-beam heating, using an apparatus similar to that first described by Calverley, Davis and Lever (14). Each rod was held vertically and a 9" section at the centre was scanned by the annular electron-gun at a speed of 3" per hour, under a vacuum of between 10^{-5} and 5×10^{-6} mm.Hg. Grain-sizes (determined by metallographic examination of at least one sample from each rod) ranging from about .002 cm to about .09 cm were obtained by using temperatures (measured with an optical pyrometer) in the range from 1420°C to 2070°C. Individual rods showed a good uniformity of grain-size, but generally differed somewhat in grain-size from other rods given an apparently similar heat-treatment; this was probably due to inaccuracies in the temperature measurements. A list of all the annealing treatments is given in Table II, and Figure 1 shows microstructures of the starting material and of typical recrystallized specimens.

Tensile test pieces were made by brazing the ends of 2" lengths of the recrystallized tantalum to threaded 5/16" dia. stainless steel collars. Carefully drilled central holes in the collars accommodated the tantalum, ensuring good alignment. Brazing was carried out in the electron-beam apparatus, at a vacuum similar to that used for the re-crystallization anneals, by melting the portion of each collar adjacent to the tantalum. Rapid wetting of the tantalum occurred with the formation of a neat, regular fillet, leaving a 1" gauge length (Figure 2). Each brazing operation took less than one minute, and metallographic examinations of numerous joints showed no grain-growth in the tantalum as a result of the brazing. The completed specimens were allowed to cool to room temperature before they were removed from the electron-beam apparatus and prior to tensile testing each one was lightly etched in a 1:1 mixture of HF and HNO_3 to allay any possible effects of surface contamination.

The tensile tests were carried out in a "hard" machine of the same basic design as that previously described by Adams (15), at temperatures of 23°C, -78°C (bath of acetone and dry-ice), and -196°C (liquid nitrogen bath). At each temperature, specimens covering the complete grain-size range were tested at two strain-rates; 1.09×10^{-3} per sec. and 9.96×10^{-2} per sec. Sometimes several specimens from a single rod were tested under the same conditions to check reproducibility but more often, in order to even out possible inaccuracies due to material variations, separate samples from the same rod were tested under different conditions. All of the stress-strain curves were autographically recorded on a milli-volt recorder whose response time (1/4 sec. for full scale) allowed the use of maximum sensitivity (10" full scale) even under the most extreme testing conditions.

To check what effects the various preparation precedures had on final specimen purity, samples which had been recrystallized at the highest, lowest and an intermediate annealing temperature, were analyzed (after tensile testing) to determine their gas contents. The results, given in Table III, show some variations from sample to sample, but the variations are small compared with the total impurity content, and they are not systematic. The latter observation suggests that they probably arise from inhomogeneities in the starting material rather than from the preparation procedures.

III. RESULTS:

A. Effect of Temperature, Strain-Rate and Grain-Size on the Form of the Stress-Strain Curve, and on the Fracture Behaviour.

Pertinent tensile and fracture examination data for all of seventy specimens tested is contained in Table IV, and Figs. 3,4, and 5 illustrate representative stress-strain curves for the various test conditions. All of the curves (except the one for a coarse grained specimen tested at -196°C at the slow strain-rate) are typical of a strain-aging material in that they show a well defined, sharp yield drop, and in their general form they are similar to curves for niobium (4) tested under like conditions. The curves show that tantalum work-hardens only slightly when deformed in tension beyond the lower yield extension, and then only when the yield stress is at a comparatively low level. Under test conditions where the (lower) yield stress is raised to above about 60,000 p.s.i. no work-hardening is observed at all; specimens tested under these conditions show local necking immediately after they yield and continue to deform at the neck under decreasing load, until they fracture.

On examining the data of Table IV and Figs. 3-5 in more detail for the influence of temperature (T), strain-rate ($\dot{\epsilon}$) and grain-size (d) on the tensile behaviour of commercial tantalum, the following general trends become apparent:

1. With $\dot{\epsilon}$ and d fixed the effect of decreasing the test temperature is to raise the upper and lower yield stresses, to raise the ultimate tensile stress (U.T.S) in specimens which work-harden, and to decrease the elongation to fracture. When the temperature is lowered from 23°C to -78°C the size of the yield drop remains essentially unaltered, but on further lowering the temperature to -196°C the yield drop on coarse grained specimens is decreased, and that on fine-grained specimens is appreciably increased.

2. With T and d fixed the effect of increasing the strain-rate at 23°C and -78°C is essentially the same as that of decreasing the temperature with strain-rate fixed; that is, the upper and lower yield stresses and the U.T.S. values are increased, the elongation to fracture is decreased, and the yield drop remains essentially constant. At -196°C a different behaviour is observed; strain-rate has, within the experimental errors, no effect on lower yield stress and elongation to fracture, and only a slight effect (increasing with increased strain-rate) on the upper yield stress at this temperature.

3. With T and $\dot{\epsilon}$ fixed the effect of decreasing the grain-size (increasing $d^{-1/2}$) at 23°C and -78°C is to raise the upper and lower yield stresses (about equally, so that the yield drop stays essentially constant) and the U.T.S. values, where these are observed. The values of elongation to fracture increase as 2d is lowered from .09cm. ($d^{-1/2} \simeq 5$cm.$^{-1/2}$) to about .0055 cm. ($d^{-1/2} \simeq 19$cm.$^{-1/2}$), and then decrease as 2d is lowered further. Changes of elongation to fracture with grain-size become less pronounced as the temperature is decreased or the strain-rate increased (Fig.6.). At -196°C decreasing values of 2d down to about .0055 cm. result in increases of both upper and lower yield stress, with the upper yield stress increasing at the faster rate. Further reductions in 2d beyond about .0055 cm. continue to increase the upper yield stress but the lower yield stress begins to fall. The overall effect is to produce a very large yield drop (as much as 25% of the upper yield stress) on specimens of very fine grain-size. Values of elongation to fracture remain essentially constant with changing grain-size at -196°C

Examination of the fractured regions of all the specimens was made after the tests. A cursory inspection showed pronounced necking in all cases, with area reductions of at least 90%, and fractures apparently wholly fibrous in character. Careful examination at high magnification revealed, however, that some of the fracture surfaces contained occasional, small, brightly reflecting facets. Focussing difficulties prevented detailed studies of many of the facets, but sufficient numbers could be studied in detail to allow a differentation into three types. Some (on samples tested at all three temperatures) were smooth and featureless, and probably of intercrystalline origin; others (on a few coarse grained samples tested at -78°C and 23°C) showed ill-defined markings which resembled cleavage steps; most important, the facets on some of the coarser grained ($d^{-1/2}$ values up to 14.2cm.$^{-1/2}$) specimens tested at -196°C showed clearly defined cleavage steps. An example is given in Fig. 7. This result then, corroborates the previous single report (11) thattantalum, whilst predominantly a ductile material, can, under suitable conditions, be made to cleave.

B. Effect of Temperature and Strain-Rate on σ_i and k_y

In Fig. 8. lower yield stress values are plotted as a function of $d^{-1/2}$ for each of the six test conditions. Within the experimental limits the results for the tests at 23°C and -78°C show a continuous linear increase of yield stress with increasing $d^{-1/2}$, as predicted by Eq. (2), and a dependency of yield strength on strain-rate at a given temperature and grain-size, is clearly evident. At -196°C the behaviour is different; the yield strength increases linearly with $d^{-1/2}$ for values of the latter up to about 20 cm.$^{-1/2}$, but then begins to fall as $d^{-1/2}$ is further increased. Furthermore, there is no clear separation of the results for the tests at the slow strain-rate from those at the fast strain-rate. Strain-rate, over the range measured, is apparently ineffective in changing the lower yield stress at -196°C.

The reason for the reduction of lower yield stress in the finest grained specimens at -196°C is not fully understood, but is believed to be associated with the very large upper yield stress exhibited by these specimens, and with the inability of tantalum to work-harden at -196°C. According to the theory of yielding outlined in the introduction, the upper yield point is the stress at which small, prematurely yielded (because of the local stress concentrations) zones in a specimen are able to trigger yield in adjacent grains. The latter grains then spread the deformation further into the specimen, through the action of dislocation pile-ups at their boundaries, at a stress (the lower yield stress) determined by the pile-up length and consequently by the grain-size. Consider, however, what would happen if the initial yielding pulse was very strong (large upper yield stress), and such (in excess of some critical value) that the avalanches of dislocation created by it were not arrested at the first grain-boundaries that they encountered. In this case, particularly if the material (because of its inability to work-harden) continued to deform only at the initial yield site, yield propagation would occur at a stress lower than that expected for the measured grain-size. It is suggested that the finest grained specimens tested at -196°C could have behaved in this manner. This explanation is, of course, largely phenomenological in that it does not provide reasons for the strong dependence of the upper yield stress on grain-size at -196°C. More elaborate experiments would be needed to determine these reasons, since, in conventional tensile tests of the type used in the present work, the upper yield stress can be critically affected by subtle test conditions - in particular by the presence of local stress concentration centres (externally, at scratches or at points where the cross-section varies, and internally at inclusions or the corners of grains) in the specimens.

From the results of Fig. 8 experimental values of σ_i and k_y have been estimated (Table V). Here shear stresses rather than tensile stresses (to a first approximation the shear stress is half the tensile stress) are used in accordance with Eq.(2). Within the experimental scatter (individual experimental points vary by about ± 5% from the mean) no variation in the dependence of yield stress on grain-size could be detected for the different test (combinations of temperature and strain-rate) conditions, and the parallel lines that are drawn through the experimental points in Fig.8. are those which give the best average overall fit. Thus, a constant value of k_y is reported in Table V, and it is estimated that this value may carry a possible error of about ± 50% (which is typical for experiments in which the variations of lower yield stress with grain-size is slight), or even somewhat more at -196°C where only tests on specimens with $d^{-1/2}$ values up to about 20cm.$^{-1/2}$ were considered. The possible errors in the σ_i values are estimated at about ± 5%, and within this margin no effect of strain-rate on σ_i is detected at -196°C.

The value of k_y for tantalum is somewhat higher (about 30%) than that for niobium (4) tested at a fast rate at -196°C. and is about six times smaller than that for mild steel (16) at the same temperature, or for molybdenum (8) in slow strain-rate tests at room temperature. The σ_i values for the slow strain-rate tests are some 50% higher than those for niobium (4) at 23°C and -78°C, and about 25% higher at -196°C. An increase of strain-rate of two orders of magnitude at 23°C or -78°C produces an effect on σ_i equivalent to that of reducing the test temperature by about 40°C.

C. Examination for Twinning:

Representative samples covering the complete range of testing conditions were sectioned, polished, and etched after testing, and carefully examined for deformation twins. Some markings which may have been twin bands were found in the necked regions of a few coarse-grained specimens tested at -196°C, but these markings were so heavily distorted that their unequivocal identification as twins was not possible. If twinning did occur in the tests it was sporadic and in very small amounts.

IV. DISCUSSION:

The present experiments have extended the scope of previous investigations (9)(10) of the low temperature tensile properties of tantalum by including an additional variable - the effect of grain-size. Inclusion of this variable has allowed a determination of experimental σ_i and k_y values for tantalum by means of the Petch analysis, and it is with the influence that these parameters exert on the mechanical behaviour of tantalum that this discussion will be primarily concerned.

The first point to note is that the k_y value for tantalum is close to that for niobium (4), and is small relative to that for mild steel (16) and for molybdenum (8). On the basis of Eq. (2) this means that the atmosphere locking of dislocations in tantalum is relatively slight, a result which may seem surprising in view of the fact that the stress-strain curves show well defined, sharp yield drops. It must be remembered, however, that the size of the yield drop is an unreliable quantitative measure of the strength of the dislocation locking because, as discussed in section III B., the experimental value of the upper yield point depends sensitively on specimen preparation and testing technique. Confirmation of a small k_y for tantalum has recently been independantly obtained by Gilbert et.al.(17) who used the Lüder's strain technique (1) to derive, from individual stress-strain curves, a value of $k_y = 1.2 \times 10^7$ c.g.s., which compares very favourably with the value of 1.04×10^7 c.g.s. obtained in the present experiments. Theoretical reasons also lead one to expect relatively weak dislocation locking in tantalum because, having a unit cell larger than that of the other body-centred-cubic transition metals, the material should be more easily able to accommodate interstitial solutes with a minimum of elastic distortion.

The results for the dependence of σ_i and k_y on temperature and strain-rate indicate that tantalum derives its sensitivity of yield strength to temperature and strain-rate from lattice friction (σ_i) rather than from dislocation locking (k_y) effects. This result appears to be general for the body-centred-cubic transition metals (2)(4)(7)(16) but has yet to be fully explained. General opinion, based on theoretical reasoning and on brief experimental evidence (16), favours the view that the Peierls-Nabarro force is probably responsible for the temperature and strain-rate dependent lattice friction stress in these metals. The observation that σ_i for tantalum becomes insensitive to strain-rate at very low temperatures tends to support this view. Analyzing a similar effect observed in chromium, Marcinkowski (7) has shown that the width of a Peierls dislocation (and consequently the Peierls-Nabarro force) becomes increasingly insensitive to velocity as the temperature is decreased.

The fracture studies have confirmed previous observations (9)(10) that tantalum exhibits considerable ductility under conditions (low temperature, high strain-rate and large grain-size) where other body-centred-cubic transition metals are brittle. Thus, every specimen showed at least 90% reduction in area before failure and all of the fractures were predominantly fibrous in character. At the same time, however, close examination did reveal cleavage facets of the fracture surfaces of some of the samples. We must now attempt to correlate these observations with the fracture theory described in the introduction.

If the theory is correct it should provide a self-consistent quantitative explanation for two critical results obtained in the experiments: (i) the ductile behaviour, at the yield point, of the coarsest grained specimens ($d^{-1/2} = 7.8 cm.^{-1/2}$) in the tests at -196°C; (ii) the occurrence, at -196°C, of cleavage facets on fractured specimens with $d^{-1/2}$ values $\leq 14.2 cm.^{-1/2}$ but not on specimens with $d^{-1/2}$ values $\geq 16.6 cm.^{-1/2}$. Ductile behaviour should correspond to a condition where the left hand side, $(\sigma_i d^{1/2} + k_y)\, k_y$, of Eq. (1) is less than the value of the right hand side, $\beta \mu \gamma$, and brittle behaviour to a condition where the left hand side exceeds the right hand side.

At the yield point for tests at -196°C we have experimental values of $\sigma_i = 4.26 \times 10^9$ dyne,cm.$^{-2}$ and $k_y = 1.04 \times 10^7$ c.g.s. Therefore, knowing that $d^{-1/2} = 7.8 cm.^{-1/2}$, $\beta = 1$ (uniaxial tension) and $\mu = 7.2 \times 10^{11}$ dyne.cm.$^{-2}$ (derived from known values of Young's modulus (18) and bulk modulus (19)) we deduce, from result (i), a boundary value

$$\gamma > (\sigma_i / d^{-1/2} + k_y)\, k_y / \beta\mu = 8.06 \times 10^3 \text{ erg.cm.}^{-2} \quad (3)$$

Applying this deduction to result (ii) we note that for cleavage to have occurred in specimens with $d^{-1/2}$ values of $14.2 cm.^{-1/2}$, a condition

$$(\sigma_i / d^{-1/2} + k_y)\, k_y / \beta\mu \geq \gamma > 8.06 \times 10^3 \text{ erg.cm.}^{-2} \quad (4)$$

must have existed in these specimens just prior to fracture. This means then (since the value of $d^{-1/2}$ is larger in Eq. (4) than in Eq. (3)) that the effect of plastic deformation must have been to raise σ_i and/or k_y, or to reduce β.

The theory indicates that such changes may be brought about in several different ways: (a) σ_i can be raised by work-hardening; (b) σ_i and k_y can be raised locally by the increased strain-rate at a neck, or at the head of an advancing fibrous fracture; (c) uniaxial tension may be converted locally to triaxial tension, thus changing β from unity to 1/3, in the notch at the base of a sharp neck or at a fibrous fracture front. Generally more than one of these mechanisms will operate simultaneously and a quantitative assessment of their effect becomes difficult. The present case is, however, simplified. No work-hardening was observed at -196°C, and σ_i and k_y were insensitive to strain rate at this temperature. Consequently we have only to consider the stress system effect; that is, the result of converting β from unity to 1/3.

With $\beta = 1/3$, $d^{-1/2} = 14.2 \text{cm.}^{-1/2}$, $\sigma_i = 4.26 \times 10^9$ dyne.cm.$^{-2}$, $k_y = 1.04 \times 10^7$ c.g.s. and $\mu = 7.2 \times 10^{11}$ dyne.cm.$^{-2}$, Eq. (4) gives

$$1.35 \times 10^4 \text{ erg.cm}^{-2} \geq \gamma > 8.06 \times 10^3 \text{ erg. cm.}^{-2} .$$

The theory is therefore in accord with the results if we make the reasonable assumption that triaxial stress conditions were created (probably when the specimens began to fail by fibrous fracture) in the deformed specimens immediately prior to their final failure.

Finally, from the observations that cleavage facets were absent from the fracture surfaces of specimens with $d^{-1/2}$ values $\geq$ 16.6 cm.$^{-1/2}$ deformed at -196°C, we are able to further narrow the limits on γ. We find, with $\beta = 1/3$, $d^{-1/2} = 16.6 \text{ cm.}^{-1/2}$, $\sigma_i = 4.26 \times 10^9$ dyne. cm.$^{-2}$, $k_y = 1.04 \times 10^7$ c.g.s. and $\mu = 7.2 \times 10^{11}$ dyne. cm.$^{-2}$, a value

$$\gamma > (\sigma_i/d^{-1/2} + k_y) k_y / \beta\mu = 1.16 \times 10^4 \text{ erg.cm.}^{-2} .$$

Thus, the effective surface energy for (cleavage) fracture in tantalum is given by

$$1.35 \times 10^4 \text{ erg. cm}^{-2} \geq \gamma > 1.16 \times 10^4 \text{ erg. cm.}^{-2}$$

The analysis reveals that tantalum has a value of γ in the same range as those values which have been experimentally determined for En. 2 steel (1.16×10^4 erg.cm^{-2} (20)), for niobium (1.36×10^4 erg. cm.$^{-2} \pm 15\%$ (4)) and for molybdenum (1.2×10^4 erg.cm.$^{-2}$(8)). Consequently we conclude that the greater resistance to brittleness of tantalum compared with these other materials, arises not from a surface energy effect, but rather from a relatively low value of k_y , combined with a high value of μ .

Implicit to the analysis is the conclusion that notched tensile samples ($\beta = 1/3$) with $d^{-1/2}$ values ≤ 14.2 cm.$^{-1/2}$ would have failed by cleavage at the yield point at -196°C. It is intended in future work to test such samples. An investigation by Imgram et.al. (21) has indicated a lack of notch sensitivity in tantalum. However, the specimens used by these workers had a $d^{-1/2}$ value of 16.2 cm.$^{-1/2}$, and were considerably more pure than those used in the present experiments.

Two further results of the fracture studies are of interest: firstly the occurrence of small areas of intercrystalline failure on many of the samples, and secondly the possible occurrence of cleavage on some of the coarse-grained samples tested at -78°C and 23°C. The first of these results suggests that the γ values for some of the grain boundaries in the specimens may have been lowered by local impurity segregation: the second indicates that work-hardening and/or strain rate effects may have raised σ_i and k_y at -78°C and 23°C sufficiently, when combined with the triaxial stress effect, to satisfy Eq. (1).

The experiments throw very little light on the relationship between twinning and the fracture behaviour of tantalum, except that they do indicate that cleavage is possible in the absence of profuse twinning. Other recent experiments (22) suggest that the relationship may be quite complex, since they indicate that interstitial impurity additions tend to suppress twinning in tantalum, whereas cleavage is known to be promoted by such additions. It is clear that more experiments, particularly of a direct observational type, will be needed before a clear understanding of this aspect of the brittle fracture problem can be gained.

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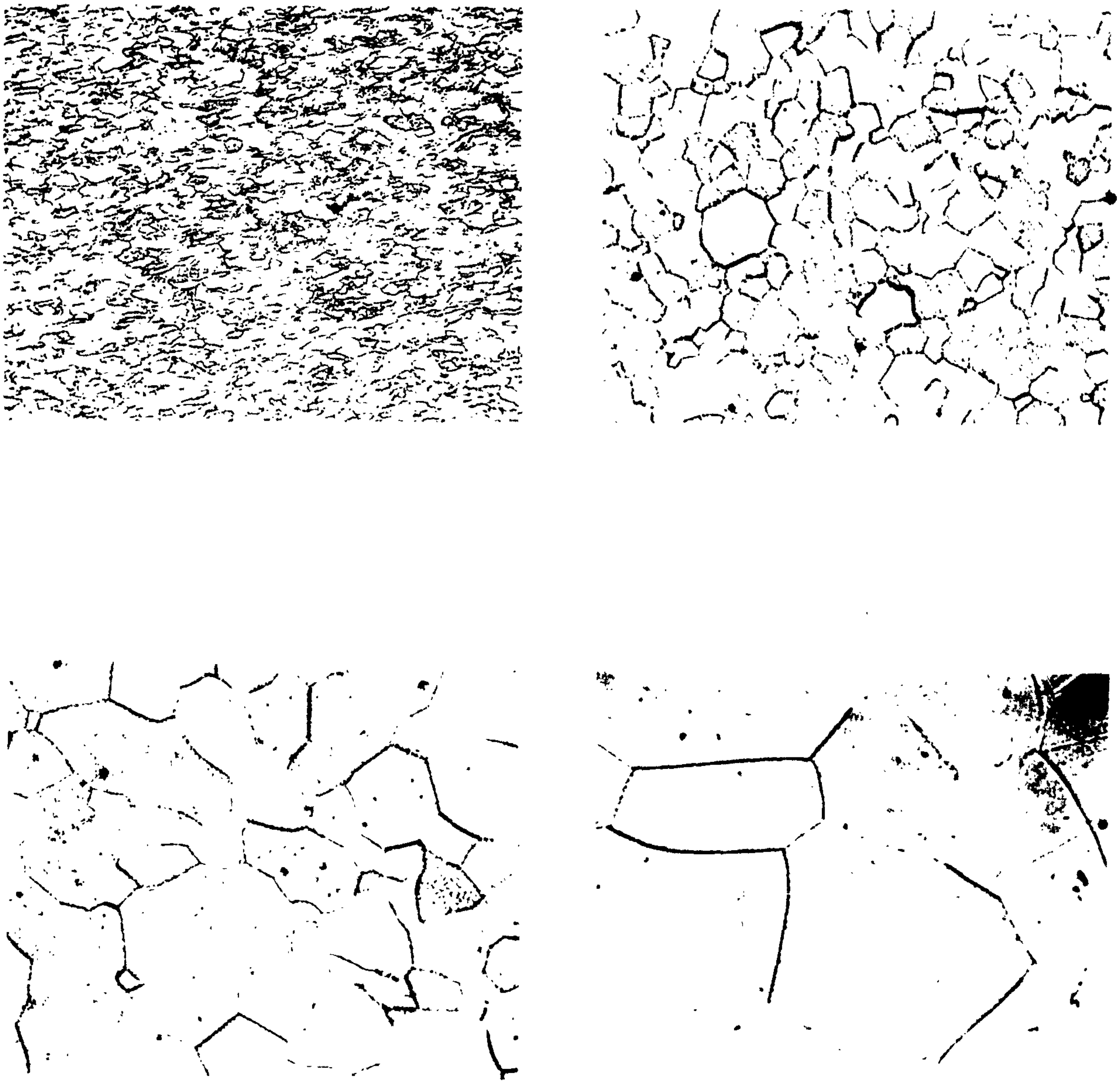

FIG. I. MICRO-STRUCTURES OF THE STARTING MATERIAL & OF TYPICAL RE-CRYSTALLIZED SPECIMENS. ETCHED 30 SECS. IN 1:1 HF/HNO_3. 200 X.

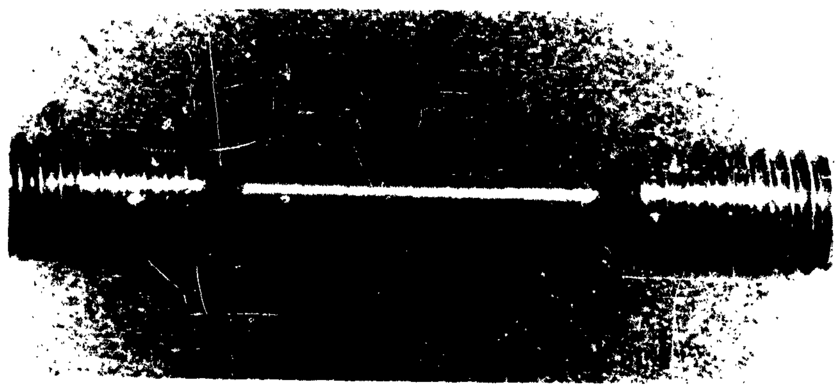

FIG. 2. TYPICAL TENSILE SPECIMEN. 2 X.

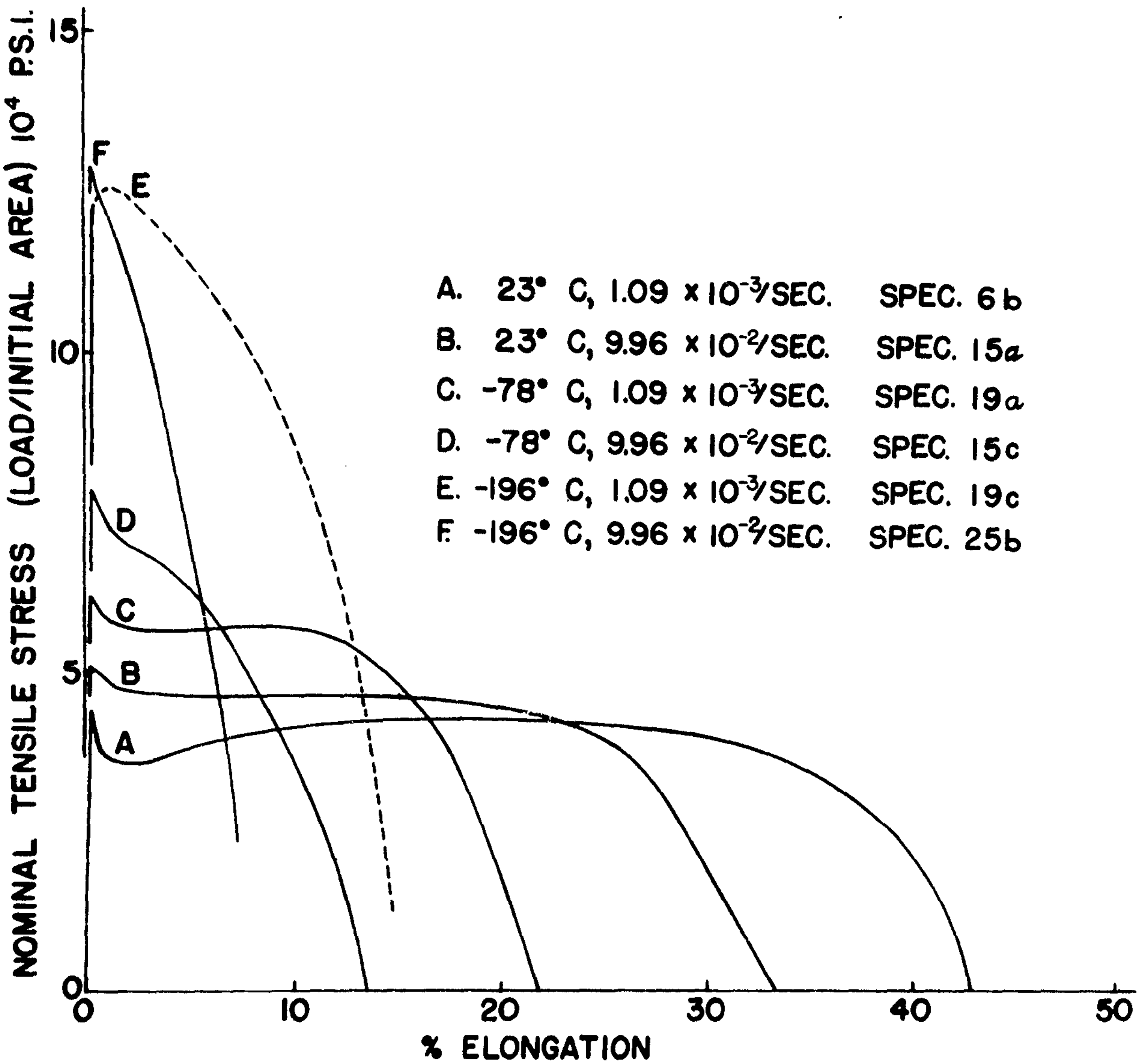

FIG. 3. EFFECT OF TEMPERATURE & STRAIN-RATE ON THE STRESS-STRAIN CURVE FOR SPECIMENS WITH $d^{-\frac{1}{2}}$ VALUES IN THE RANGE 7.8 $CM.^{-\frac{1}{2}}$ TO 10.5 $CM.^{-\frac{1}{2}}$

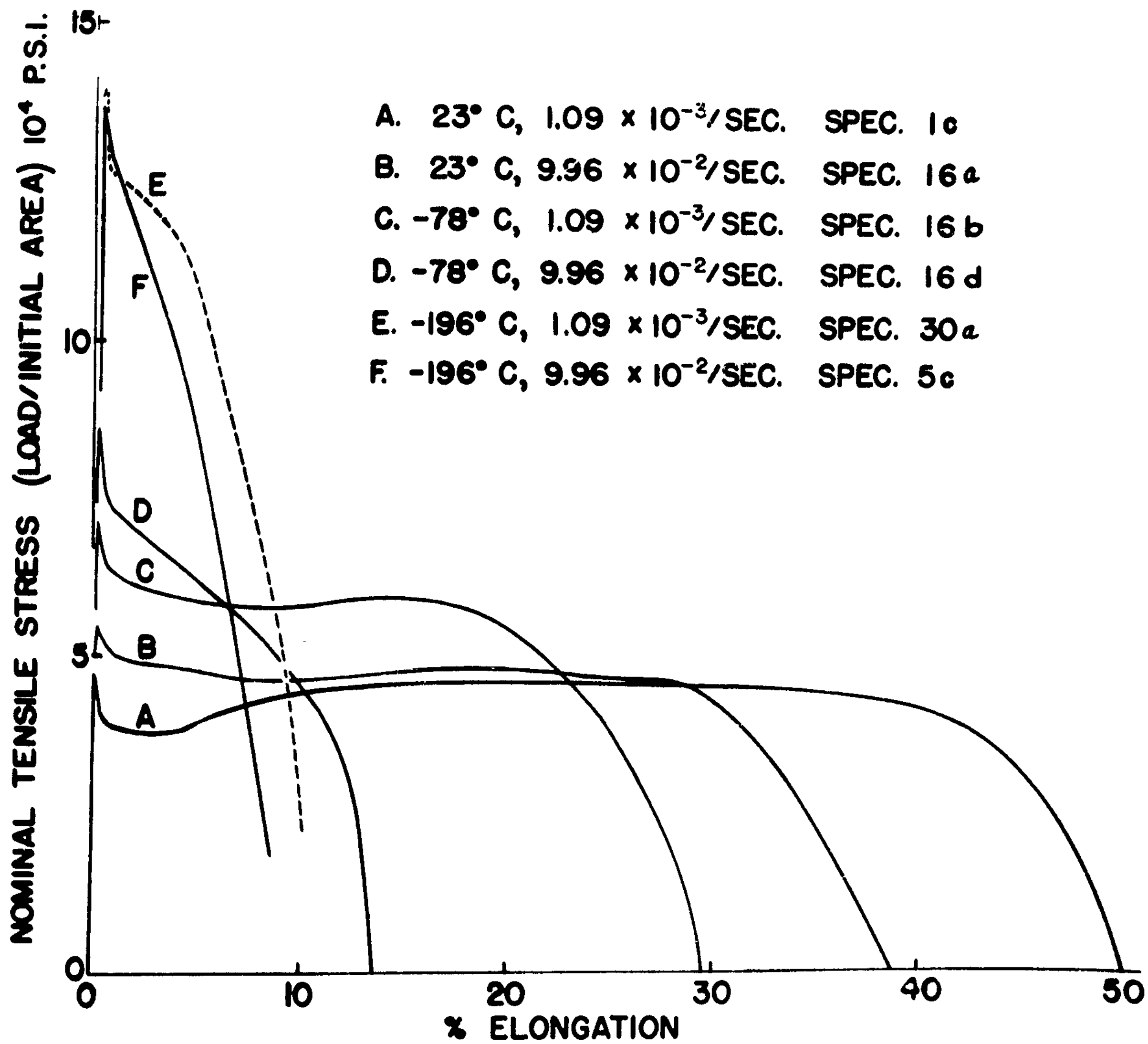

FIG. 4. EFFECT OF TEMPERATURE & STRAIN-RATE ON THE STRESS-STRAIN CURVE FOR SPECIMENS WITH $d^{-\frac{1}{2}}$ VALUES IN THE RANGE 16.6 CM.$^{-\frac{1}{2}}$ TO 18.8 CM.$^{-\frac{1}{2}}$

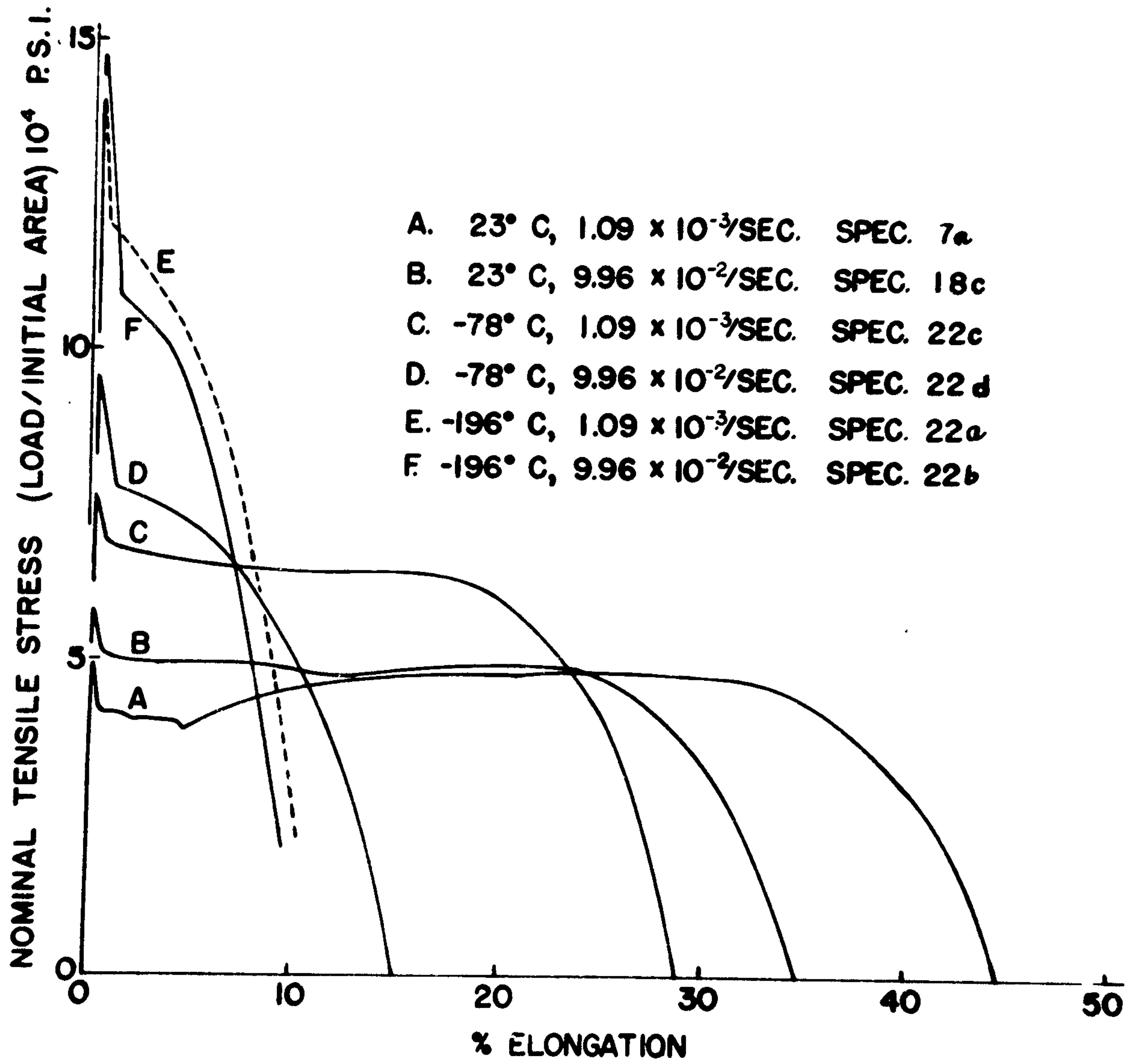

FIG. 5. EFFECT OF TEMPERATURE & STRAIN-RATE ON THE STRESS-STRAIN CURVE FOR SPECIMENS WITH $d^{-\frac{1}{2}}$ VALUES IN THE RANGE 23.5 $CM^{-\frac{1}{2}}$ TO 30.2 $CM^{-\frac{1}{2}}$

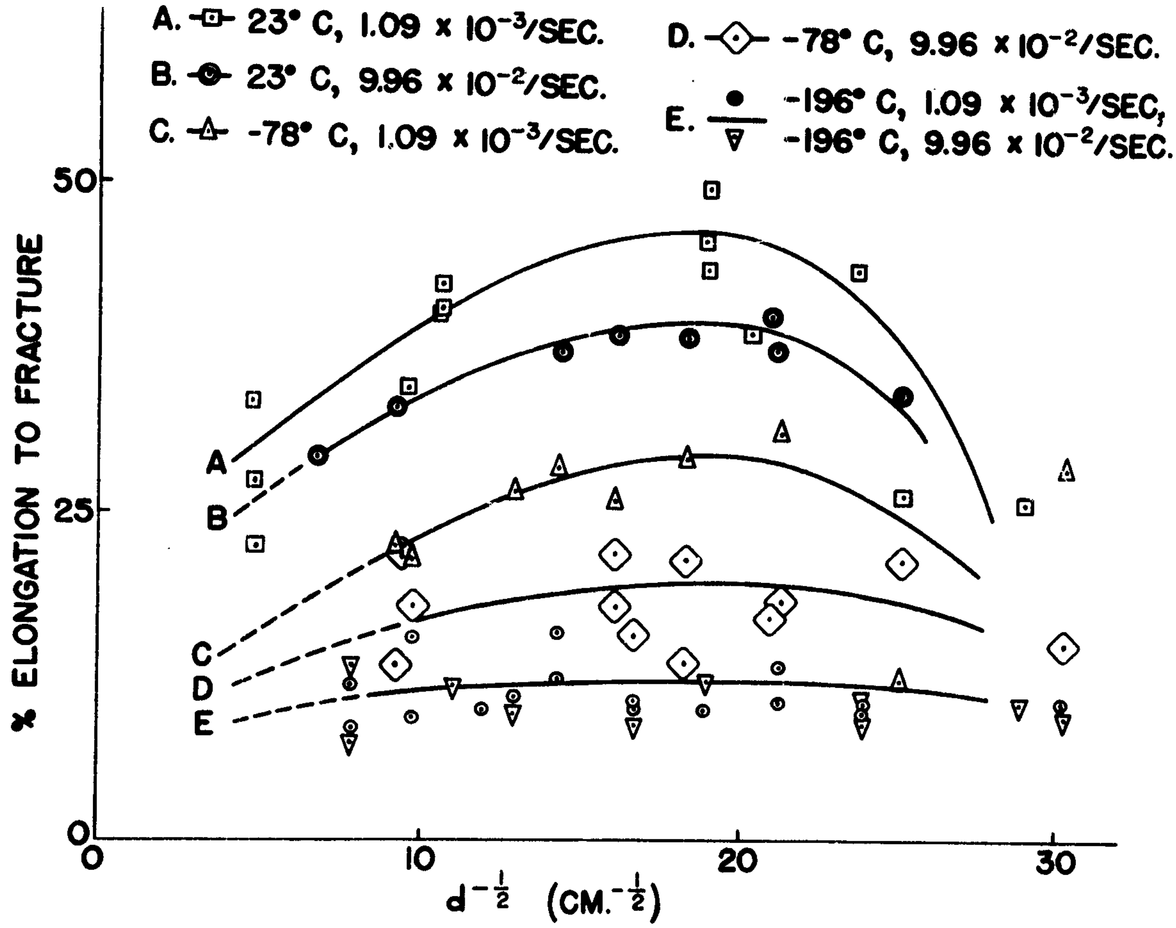

FIG. 6. EFFECT OF TEMPERATURE, STRAIN-RATE, & GRAIN-SIZE (2d) ON THE ELONGATION TO FRACTURE.

FIG. 7. CLEAVAGE FACET ON A SPECIMEN (19c) FRACTURED AT -196° C 600 X.

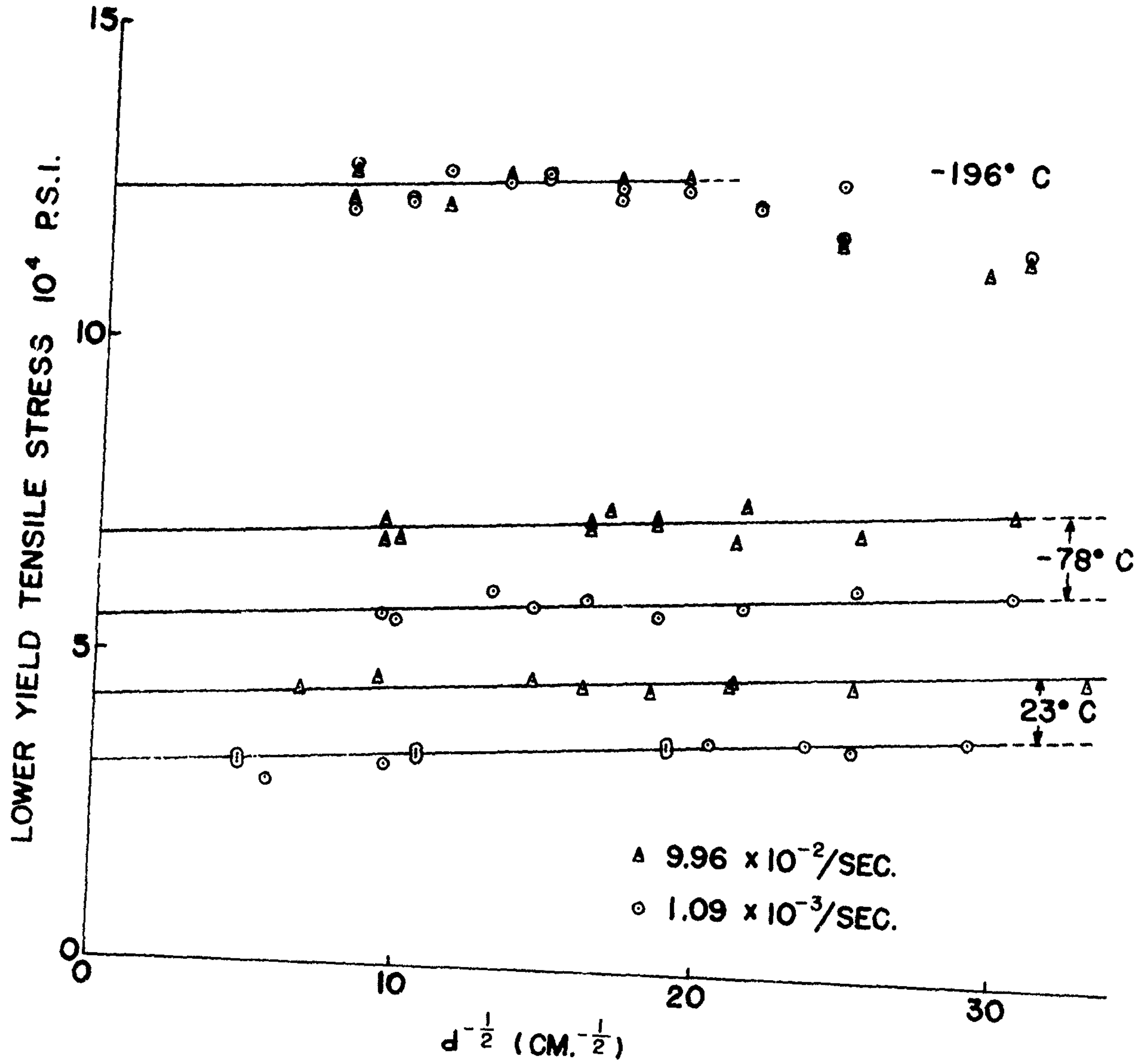

FIG. 8. EFFECT OF TEMPERATURE & STRAIN-RATE ON THE VARIATION OF LOWER YIELD STRESS WITH GRAIN-SIZE (2d).

TABLE I

NOMINAL ANALYSIS OF THE "AS-SUPPLIED" TANTALUM

Impurity Element	O_2	N_2	H_2	C	Cb	Fe	Ti	Si	Al
p.p.m. present	150	100	2	50	<100	50	30	200	30

TABLE II

ANNEALING TREATMENTS FOR RE-CRYSTALLIZATION

Annealing Temperature °C	Grain-sizes Obtained on Different Rods 2d(cm.)	$d^{-1/2}$(cm.$^{-1/2}$)
1420	.0049 to .0019	20.2 to 32.8
1560	.0060 to .0035	18.2 to 23.9
1810	.0182 to .0078	10.5 to 16.0
2070	.0889 to .0212	4.7 to 9.7

TABLE III

GAS CONTENTS OF THE RE-CRYSTALLIZED MATERIALS

Annealing Temperature °C	Gas Content (p. p. m.) O_2	N_2	H_2
1420	81	46	<0.5
1810	87	101	<0.5
2070	79	41	8

TABLE IV
TENSILE PROPERTIES AND FRACTURE BEHAVIOUR OF
COMMERCIAL TANTALUM

Spec. No.*	Temp °C	Strain Rate sec^{-1}	$d^{-1/2}$ $cm^{-1/2}$	Upper Yield Stress p.s.i.	Lower Yield Stress p.s.i.	Ultimate Tensile Stress p.s.i.	Uniform Elong. %	Elong. to Fracture %	Fracture Behaviour**
4a	23	1.09×10^{-3}	4.7	38,296	32,801	35,354	11.79	27.60	A,1.
4b	23	1.09×10^{-3}	4.7	40,849	33,044	36,181	9.29	22.63	A,3.
4c	23	1.09×10^{-3}	4.7	37,810	33,068	36,716	13.85	33.64	A,2.
8b	23	1.09×10^{-3}	9.5	40,687	32,579	36,997	14.27	34.76	A,4.
6a	23	1.09×10^{-3}	10.5	40,849	34,868	40,898	21.06	40.45	A,4.
6b	23	1.09×10^{-3}	10.5	44,372	35,473	42,019	16.55	42.75	A,2.
6c	23	1.09×10^{-3}	10.5	41,627	35,403	41,433	15.77	40.87	A,2.
1a	23	1.09×10^{-3}	18.0	45,932	38,526	47,097	23.57	44.25	B,1.
1c	23	1.09×10^{-3}	13.5	47,005	37,422	45,967	23.05	50.13	B,2.
1d	23	1.09×10^{-3}	18.0	38,446	37,154	46,058	22.94	46.42	B,2.
2a	23	1.09×10^{-3}	20.2	46,211	38,324	47,821	22.42	39.14	C,1.
7a	23	1.09×10^{-3}	23.5	49,893	38,942	48,600	27.62	44.28	C,1.
18b	23	1.09×10^{-3}	25.0	41,985	38,130	47,298	17.00	26.[illegible]4	C,3.
26b	23	1.09×10^{-3}	28.9	45,631	40,337	46,152	14.02	26.12	C,3.
12d	23	9.96×10^{-2}	6.7	52,910	44,919	45,194	8.56	29.46	A,4.
15a	23	9.96×10^{-2}	9.7	50,816	46,902	46,902	11.20	33.15	A,2.
9d	23	9.96×10^{-2}	14.3	53,006	47,610	47,610	11.87	37.70	B,1.
13a	23	9.96×10^{-2}	16.0	58,376	46,929	47,438	12.12	39.05	B,2.
16a	23	9.96×10^{-2}	18.2	54,389	46,500	47,659	17.33	38.81	B,1.
14a	23	9.96×10^{-2}	20.9	59,017	48,295	48,968	18.05	40.57	C,3.
11d	23	9.96×10^{-2}	21.0	56,417	48,652	48,810	13.80	37.88	B,1.
1[illegible]c	23	9.96×10^{-2}	25.0	58,404	48,339	49,878	20.02	34.78	C,3.
10d	23	9.96×10^{-2}	32.8	51,792	50,724	52,439	-	-	-

* Specimens with the same no. were taken from the same recrystallized rod.

** A.-Ragged shear. B.-Chisel edge. C.-Cup and cone.
1.-No shiny facets detected. 2.-Some shiny facets of indeterminate surface topography.
3.-Shiny facets with smooth surfaces. 4.-Shiny facets with possible cleavage steps
5.-Shiny facets with cleavage steps.

TABLE IV (Cont.)

Spec. No.	Temp. °C	Strain Rate sec^{-1}	$d^{-1/2}$ $cm^{-1/2}$	Upper Yield Stress p.s.i.	Lower Yield Stress p.s.i.	Ultimate Tensile Stress p.s.i.	Uniform Elong. %	Elong. to Fracture %	Fracture Behaviour
15b			9.2	64,938	57,071	51,623	7.37	22.71	A,2.
19a			9.7	61,839	56,448	57,141	8.02	21.74	A,4.
24d			12.9	72,575	61,964	61,964	12.84	26.79	B,1.
17a			14.2	71,892	59,772	61,730	10.67	28.99	B,3.
13b	-78	1.09 $X10^{-3}$	16.0	67,404	61,145	62,590	7.78	26.25	B,2.
16b			18.2	71,817	58,944	58,944	12.87	29.52	B,1.
20a			21.2	72,269	60,751	62,257	12.58	31.68	B,2.
18a			25.0	72,323	64,506	-	-	12.59	C,2.
22c			30.2	76,861	64,400	-	-	28.89	C,2.
15c			9.2	78,745	72,751	-	-	13.40	A,2.
15d			9.2	73,023	69,527	-	-	22.06	A,4.
19d			9.7	75,583	69,240	-	-	17.82	A,3.
13c			16.0	81,432	73,681	-	-	17.94	B,3.
13d			16.0	80,013	72,457	-	-	21.89	B,3.
5d	-78	9.96 $X10^{-2}$	16.6	84,856	75,599	-	-	15.72	B,2.
16c			18.2	78,954	74,647	-	-	21.59	B,1.
16d			18.2	86,224	74,217	-	-	13.59	B,1.
14c			20.9	79,110	71,217	-	-	17.18	C,1.
20d			21.2	82,394	77,425	-	-	18.09	B,2.
18d			25.0	76,906	73,462	-	-	21.94	C,3.
22d			30.2	95,350	77,723	-	-	15.22	C,2.

TABLE IV (Cont.)

Spec. No.	Temp. °C	Strain Rate sec^{-1}	$d^{-1/2}$ $cm^{-1/2}$	Upper Yield Stress p.s.i.	Lower Yield Stress p.s.i.	Ultimate Tensile Stress p.s.i.	Uniform Elong. %	Elong. to Fracture %	Fracture Behaviour
25a			7.8	-	122,036	-	-	8.66	A,5
25d			7.8	-	129,386	-	-	12.04	A,5.
19b			9.7	126,009	123,472	-	-	9.56	A,2.
19c			9.7	125,540	124,185	-	-	15.71	A,5.
21b			10.9	-	128,751	-	-	10.31	B,4.
24a			12.9	133,611	127,658	-	-	11.16	B,5.
17b			14.2	134,438	129,642	-	-	16.13	B,5.
17c			14.2	133,445	128,981	-	-	12.55	B,2.
5a			16.6	135,690	127,728	-	-	11.00	B,2.
5b	-196	1.09	16.6	132,538	125,239	-	-	10.59	B,2.
3a		$X10^{-3}$	18.8	140,391	127,823	-	-	10.12	B,2.
20b			21.2	128,319	125,260	-	-	13.51	B,1.
20c			21.2	133,978	125,367	-	-	10.79	B,1.
23a			23.9	134,310	121,558	-	-	10.71	C,3.
27b			23.9	138,108	129,359	-	-	10.06	B,1.
22a			30.2	140,225	120,051	-	-	10.55	C,3.
25b			7.8	128,815	124,020	-	-	7.45	A,5.
25c			7.8	-	128,059	-	-	13.44	A,1.
21a			10.9	136,843	123,258	-	-	11.84	B,4.
24b	-196	9.96	12.9	140,934	129,010	-	-	9.94	B,5.
5c			16.6	138,510	128,723	-	-	8.85	B,2.
3b		$X10^{-2}$	18.8	132,784	129,973	-	-	12.23	B,2.
23b			23.9	136,959	120,730	-	-	9.11	C,1.
27a			23.9	152,462	121,043	-	-	10.92	B,1.
26a			28.9	138,377	117,416	-	-	10.84	C,1.
22b			30.2	147,227	118,742	-	-	9.74	C,1.

TABLE V

EFFECT OF TEMPERATURE & STRAIN-RATE ON THE σ_i & k_y VALUES FOR COMMERCIAL TANTALUM.

Temperature °C	Strain-rate $sec.^{-1}$	σ_i dyne. $cm^{-2} \times 10^9$	k_y c.g.s.$\times 10^7$
23	1.09×10^{-3}	1.08	
23	9.96×10^{-2}	1.45	
-78	1.09×10^{-3}	1.90	1.04
-78	9.96×10^{-2}	2.35	
-196	1.09×10^{-3}	4.26	
-196	9.96×10^{-2}		